

TRANSMITTAL LETTER OF THE UNITED STATES DESIGNATED/ELECTED OFFICE (DO/EO/US) CONCERNING A FILING UNDER 35 U.S.C. 371		Attorney Docket No. 0502-1002 U.S. Application No. 10/070173
INTERNATIONAL APPLN. NO. PCT/FR00/02471	INTERNATIONAL FILING DATE 07 SEPTEMBER 2000 (07.09.00)	PRIORITY DATE CLAIMED 07 SEPTEMBER 1999 (07.09.99)
TITLE OF INVENTION: METHOD AND DEVICE FOR CONTINUOUS TREATMENT OF COPPER SULPHIDE CONTAINING ORE BY BIOLOGICAL LEACHING		
APPLICANT(S) FOR DE/EO/US: DOMINIQUE MORIN, PAUL NORRIS, CHRISTOPHER BONNEY, WERNER SCHWAB AND DIETGER KÖPPL		
Applicant herewith submits to the United States Designated Elected Office (DO/EO/US) the following items and other information:		
<ol style="list-style-type: none"> 1. <input checked="" type="checkbox"/> This is a FIRST submission of items concerning a filing under 35 U.S.C. 371. 2. <input type="checkbox"/> This is a SECOND or SUBSEQUENT submission of items concerning a filing under 35 U.S.C. 371. 3. <input checked="" type="checkbox"/> This is an express request to begin national examination procedures (35 U.S.C. 371(f)). The submission must include items (5), (6), (9) and (21) indicated below. <ol style="list-style-type: none"> <input checked="" type="checkbox"/> The US has been elected by the expiration of 19 months from the priority date (Article 31). <input checked="" type="checkbox"/> A copy of the International Application as filed (35 U.S.C. 371 (c)(2)) <ol style="list-style-type: none"> a. <input checked="" type="checkbox"/> is attached hereto (required only if not communicated by the International Bureau) b. <input checked="" type="checkbox"/> has been communicated by the International Bureau. See attached PCT/IB/308. c. <input type="checkbox"/> is not required, as the application was filed in the United States Receiving Office (RO/US). <input checked="" type="checkbox"/> An English language translation of the International Application as filed (35 U.S.C. 371 (c)(2)) <ol style="list-style-type: none"> a. <input checked="" type="checkbox"/> is attached hereto. b. <input type="checkbox"/> has been previously submitted under 35 U.S.C. 154(d)(4). <input type="checkbox"/> Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371 (c)(3)) <ol style="list-style-type: none"> a. <input type="checkbox"/> are attached hereto (required only if not communicated by the International Bureau). b. <input type="checkbox"/> have been communicated by the International Bureau. c. <input type="checkbox"/> have not been made, however, the time limit for making such amendments has NOT expired. d. <input type="checkbox"/> have not been made and will not be made. 8. <input type="checkbox"/> An English language translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371 (c)(3)). 9. <input checked="" type="checkbox"/> An oath or declaration of the inventor(s) (35 U.S.C. 371(c)(4)). 10. <input type="checkbox"/> An English language translation of the annexes of the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371(c)(5)). 		
Items 11 to 20 below concern document(s) or information included:		
<ol style="list-style-type: none"> 11. <input checked="" type="checkbox"/> Information Disclosure Statement (IDS) w/PTO-1449 - <input checked="" type="checkbox"/> Copy of IDS citations 12. <input checked="" type="checkbox"/> Assignment Papers (cover sheet & document(s)) 13. <input checked="" type="checkbox"/> A FIRST Preliminary Amendment. 14. <input type="checkbox"/> A SECOND or SUBSEQUENT Preliminary Amendment. 15. <input type="checkbox"/> A substitute specification. 16. <input type="checkbox"/> A change of power of attorney and/or address letter. 17. <input type="checkbox"/> A computer-readable form of the sequence listing in accordance with PCT Rule 18. <input type="checkbox"/> A second copy of the published international application under 35 U.S.C. 154(d)(4). 19. <input type="checkbox"/> A second copy of the English language translation of the international application (35 U.S.C. 154(d)(4)). 20. <input checked="" type="checkbox"/> Other items or information: INTERNATIONAL PRELIMINARY EXAMINATION REPORT (PCT/PEPA/409), INTERNATIONAL SEARCH REPORT (PCT/ISA/210), APPLICATION DATA SHEET, ABSTRACT 		

U.S. APPLICATION NO. 107070173		INTERNATIONAL APPLN. NO. 07 SEPTEMBER 2000 (07.09.00)		ATTORNEY DOCKET NO. 0502-1002	
21. <input checked="" type="checkbox"/> The following fees are submitted:				CALCULATIONS PTO USE ONLY	
BASIC NATIONAL FEE (37 CFR 1.492 (a) (1)-(5):					
Neither international preliminary examination fee nor international search fee paid to USPTO and international Search Report not prepared by the EPO or JPO.....\$1040.00					
International preliminary examination fee not paid to USPTO but International Search Report prepared by the EPO or JPO\$890.00					
International preliminary examination fee not paid to USPTO but International search fee paid to USPTO\$740.00					
International preliminary examination fee paid to USPTO but all claims did not satisfy provision of PCT Article 33 (1)-(4)\$710.00					
International preliminary examination fee paid to USPTO and all claims satisfied provision of PCT Article 33 (1)-(4)\$100.00					
ENTER APPROPRIATE BASIC FEE AMOUNT				\$ 890.00	
Surcharge of \$130.00 for furnishing the oath or declaration than <input type="checkbox"/> 20- <input type="checkbox"/> 30 months from the earliest claimed priority date (37 CFR 1.492(e))				\$	
CLAIMS	NUMBER FILED	NUMBER EXTRA	RATE		
Total Claims	13 - 20 =	0	X \$18.00	\$	
Independent Claims	2 - 3 =	0	X \$84.00	\$	
MULTIPLE DEPEND CLAIM(S) (if applicable)			+ \$280.00	\$	
TOTAL OF ABOVE CALCULATION -				\$ 890.00	
<input type="checkbox"/> Applicant claims small entity status. See 37 CFR 1.27. The fees indicated above are reduced by 1/2.				\$	
SUBTOTAL =				\$ 890.00	
Processing fee of \$130.00 for furnishing the English translation later than <input type="checkbox"/> 20 <input type="checkbox"/> 30 months from the earliest claimed priority date (37 CFR 1.492Z(f)).				\$	
TOTAL NATIONAL FEE =				\$ 890.00	
Fee for recording the enclosed assigned (37 CFR 1.21(h)). The assignment must be accompanied by an appropriate cover sheet (37 CFR 3.28, 3.31) \$40.00 per property +				\$ 40.00	
TOTAL FEES ENCLOSED -				\$ 930.00	
				Amount to be refunded:	\$
				Charged:	\$
<input checked="" type="checkbox"/> A Check in the amount of \$930.00 to cover all fees is attached. <input type="checkbox"/> The Commissioner is hereby authorized to charge indicated fees and credit any overpayments to Deposit account No. 25-0120 in the name of Young & Thompson, as described below. A duplicate copy of this sheet is enclosed. <input checked="" type="checkbox"/> The Commissioner is hereby authorized in this, concurrent, and future replies, to charge payment or credit any overpayment to Deposit Account No. 25-0120 for any additional fee required under 37 C.F.R. §§ 1.16 or 1.17.					
SEND ALL CORRESPONDENCE TO: 745 South 23 rd Street Arlington, VA 22202 Telephone (703) 521-2297 Y&T Customer No. 000466				SIGNATURE <u><i>Benoit Castel</i></u> NAME <u>Benoit Castel</u>	
BC/bam Date: March 4, 2002				35,041 REGISTRATION NO.	

PATENT
0502-1002**IN THE U.S. PATENT AND TRADEMARK OFFICE**

In re application of: Dominique MORIN et al.

Appl. No.: **NEW NATIONAL PHASE
APPLICATION IN THE
UNITED STATES**

Group:

Filed: March 4, 2002

Examiner:

For: METHOD AND DEVICE FOR CONTINUOUS
TREATMENT OF COPPER SULPHIDE CONTAINING
ORE BY BIOLOGICAL LEACHING**PRELIMINARY AMENDMENT**Assistant Commissioner for Patents
Washington, DC 20231

March 4, 2002

Sir:

Prior to the first Official Action and calculation of the filing fee, the following preliminary amendments and remarks are respectfully submitted in connection with the above-identified application.

IN THE ABSTRACT OF THE DISCLOSURE:

Please replace the Abstract of the Disclosure with the rewritten Abstract of the Disclosure attached on a separate sheet attached hereto.

IN THE CLAIMS:

Please substitute claims 1-3 as originally filed, which appear on page 15, with claims 1-3 as filed in the Article 34 amendment of November 22, 2001. The page containing claims 1-3 is marked "AMENDED SHEET" and is attached hereto. Following the insertion of claims 1-3, please amend the claims as follows:

Please amend the claims as follows:

--3. (Amended) Method according to Claim 1, characterized in that the bacterial culture used was previously subjected to an adaptation, by successive transplants on a substrate, particularly of chalcopyrite, by progressively and artificially increasing the concentrations of the copper in solution, in order to render it able to develop in media of which the mass concentrations of copper are of the order of 50 g/l.

4. (Amended) Method according to Claim 1, characterized in that a pH included between 1.2 and 1.6 is maintained in the biological leaching reactors (1).

6. (Amended) Method according to Claim 1, characterized in that all along the phase of biological leaching, the physiological state of the bacterial culture is monitored with the aid of means for on-line analysis of the gases emerging from the reactors.

7. (Amended) Method according to Claim 1, characterized in that the step of biological leaching is followed by a second step during which, in a first phase:

- the pulp issuing from the biological leaching reactors (1) is admitted in precipitation reactors (5) in which the iron is eliminated by provoking a precipitation of jarosite, by addition of calcite, and the solution is maintained at a pH less than 3,

- the neutralized pulp is admitted in a decanter (7) and a part of the solids is made to recirculate at the head of the precipitation reactors (5).

10.(Amended) Method according to Claim 7, characterized in that the outlet (19) of the extraction unit (17) is placed in communication with the inlet (4) of the precipitation reactors (5), so as to cause part of the raffinate collected at this outlet (19) to be recirculated by causing it to traverse said reactors (5) again with a high flowrate (Q2) with respect to the flowrate (Q1) of the pulp coming from the biological leaching reactors (1), so as to provoke a dilution of the aqueous solution subjected to extraction, up to a concentration of copper of the order of about 10 g/l, i.e. up to a value corresponding to the possible extraction of the copper in an extraction unit (17).--

Please add the following claim:

--13.(New) Method according to Claim 2, characterized in that the bacterial culture used was previously subjected to an adaptation, by successive transplants on a substrate, particularly of chalcopyrite, by progressively and artificially increasing the concentrations of the copper in solution, in order to render it able to develop in media of which the mass concentrations of copper are of the order of 50 g/l.--

REMARKS

Claim 13 has been added.

Claims 3-4, 6-7 and 10 have been amended to eliminate multiple dependencies.

The substitution of claims 1-3 has been done to merely place this national phase application in into the same condition as it was during Chapter II of the International Phase.

Entry of the above amendments is earnestly solicited. An early and favorable first action on the merits is earnestly requested.

Should there be any matters that need to be resolved in the present application, the Examiner is respectfully requested to contact the undersigned at the telephone number listed below.

Attached hereto is a marked-up version of the changes made to the claims and abstract by the current amendment. The attached page is captioned "VERSION WITH MARKINGS TO SHOW CHANGES MADE."

The Commissioner is hereby authorized in this, concurrent, and future replies, to charge payment or credit any overpayment to Deposit Account No. 25-0120 for any additional fees required under 37 C.F.R. § 1.16 or under 37 C.F.R. § 1.17.

Respectfully submitted,

YOUNG & THOMPSON



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BC/bam
Attachments

VERSION WITH MARKINGS TO SHOW CHANGES MADEIN THE ABSTRACT OF THE DISCLOSURE:

The Abstract of the Disclosure has been amended as follows:

METHOD AND DEVICE FOR CONTINUOUS TREATMENT OF COPPER SULPHIDE
CONTAINING ORE BY BIOLOGICAL LEACHING

ABSTRACT

~~The invention concerns a~~ method and a device for treating copper sulphide containing ore, ~~comprising~~includes a step of biological leaching whereby the minerals are subjected in reactors (1) in cascade arrangement, wherein the temperature is maintained between 75°C and 85°C to the action of a bacterial culture, which ~~comprises~~includes a thermophilic bacterium of the Sulfolobus type, leading to solution heat treating of the copper. The method is characterized in that, during ~~said~~the biological leaching step, the treatment is uninterrupted, the medium containing the bacterial culture being continuously mechanically agitated to ensure oxygenation thereof, and suspension of the solid elements, and the solid mass proportion of the culture medium is maintained above 10%.

Translator's Note: ~~The following legend appears in the single Figure:-~~

FIRST STEP — BIOLOGICAL LEACHING

SECOND STEP — PRECIPITATING IRON
ELIMINATING IRON IN SOLUTION AND FILTERING
EXTRACTION

THIRD STEP — ELECTROLYSIS

IN THE CLAIMS:

The claims have been amended as follows:

3. (Amended) Method according to ~~one of the preceding Claims~~, Claim 1, characterized in that the bacterial culture used was previously subjected to an adaptation, by successive transplants on a substrate, particularly of chalcopyrite, by progressively and artificially increasing the concentrations of the copper in solution, in order to render it able to develop in media of which the mass concentrations of copper are of the order of 50 g/l.

4. (Amended) Method according to ~~one of the preceding Claims~~, Claim 1, characterized in that a pH included between 1.2 and 1.6 is maintained in the biological leaching reactors (1).

6. (Amended) Method according to ~~one of the preceding Claims~~, Claim 1, characterized in that all along the phase of biological leaching, the physiological state of the bacterial culture is monitored with the aid of means for on-line analysis of the gases emerging from the reactors.

7. (Amended) Method according to ~~one of the preceding Claims~~, Claim 1, characterized in that the step of biological leaching is followed by a second step during which, in a first phase:

- the pulp issuing from the biological leaching reactors (1) is admitted in precipitation reactors (5) in which the iron is eliminated by provoking a precipitation of jarosite, by addition of calcite, and the solution is maintained at a pH less than 3,
- the neutralized pulp is admitted in a decanter (7) and a part of the solids is made to recirculate at the head of the precipitation reactors (5).

10. (Amended) Method according to ~~one of Claims 7 to 9, Claim 7,~~ characterized in that the outlet (19) of the extraction unit (17) is placed in communication with the inlet (4) of the precipitation reactors (5), so as to cause part of the raffinate collected at this outlet (19) to be recirculated by causing it to traverse said reactors (5) again with a high flowrate (Q2) with respect to the flowrate (Q1) of the pulp coming from the biological leaching reactors (1), so as to provoke a dilution of the aqueous solution subjected to extraction, up to a concentration of copper of the order of about 10 g/l, i.e. up to a value corresponding to the possible extraction of the copper in an extraction unit (17).

CLAIMS

1. Method for treating copper sulphide containing ore, comprising a step of
5 biological leaching whereby the minerals are subjected, in reactors (1) in cascade arrangement, wherein the temperature is maintained between 75°C and 85°C, to the action of a bacterial culture, which comprises a thermophilic bacterium of the Sulfolobus type, leading to solution of the copper, in which a solid mass proportion of the culture medium is maintained above 10%, characterized in that,
10 during said biological leaching step:
- a continuous supply of the sulphur containing minerals is effected,
 - the medium containing the bacterial culture is subjected to continuous mechanical rotary stirring to ensure oxygenation thereof and suspension of the solid elements,
- 15 - oxygenation is promoted by an injection into the medium of air enriched with pure oxygen.
2. Method according to Claim 1, characterized in that the sulphur containing minerals are supplied to the culture medium in the form of a sulphur containing concentrate presenting a granulometry d80 less than one hundred micrometers.
- 20 3. Method according to one of the preceding Claims, characterized in that the bacterial culture used was previously subjected to an adaptation, by successive transplants on a substrate, particularly of chalcopyrite, by progressively and artificially increasing the concentrations of the copper in solution, in order to render it able to develop in media of which the mass concentrations of copper are
25 of the order of 50 g/l.

METHOD AND DEVICE FOR CONTINUOUS TREATMENT OF COPPER SULPHIDE
CONTAINING ORE BY BIOLOGICAL LEACHING

ABSTRACT

A method and a device for treating copper sulphide containing ore, includes a step of biological leaching whereby the minerals are subjected in reactors (1) in cascade arrangement, wherein the temperature is maintained between 75°C and 85°C to the action of a bacterial culture, which includes a thermophilic bacterium of the Sulfolobus type, leading to solution heat treating of the copper. The method is characterized in that, during the biological leaching step, the treatment is uninterrupted, the medium containing the bacterial culture being continuously mechanically agitated to ensure oxygenation thereof, and suspension of the solid elements, and the solid mass proportion of the culture medium is maintained above 10%.

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METHOD AND DEVICE FOR CONTINUOUS TREATMENT OF COPPER
SULPHIDE CONTAINING ORE BY BIOLOGICAL LEACHING

The present invention relates to a method and a device for continuously
5 treating copper sulphide containing ores with a view to recovering said copper.

Numerous metals, such as copper, silver, gold or palladium, exist in nature
in association with other ores. In order to recover them, many methods have been
described in the prior state of the art, consisting, during a first step of biological
leaching, in causing to act on ores bacteria of diverse type which ensure
10 destruction of the sulphur-containing matrix imprisoning a determined metal, by
dissolution of this matrix, this having the effect of dissolving the metal in
question. In a second step of treatment, the metal contained in this solution is
recovered by intervening on said solution, particularly by the chemical and/or
electrochemical route.

15 For example, US-A-4 571 387 proposed a method of leaching sulphurous
copper ores, for example chalcopyrite (CuFeS_2), in which the copper ores are
placed in contact with strains of the bacterium *Thiobacillus ferrooxydans* capable
of oxidizing the sulphides, in an aqueous acid solution, and Cu^{2+} ions as well as
sulphur and sulphate or sulphuric acid form by oxidation of the ore. The Cu^{2+} ions
20 may then be treated by a liquid/liquid extraction. A similar method is described in
U.S. Patent No. 4 729 788 which describes the use of

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thermophilic bacteria of *Sulfolobus* type to ensure leaching of sulphurous gold and silver ores.

U.S. Patent 5 919 674 also proposed a method for carrying out a part of the first step of continuous biological leaching of copper ores by means of a bacterium, particularly of *Sulfolobus* type. However, it appears that such a method does not easily lend itself to implementation from the industrial standpoint.

The present invention has for its object to propose a method aiming at recovering the copper contained in sulphur containing ores, comprising a step of continuous biological leaching, and this by using a novel thermophilic bacterial culture, of *Sulfolobus* type.

The present invention thus has for its object a method for treating copper sulphide containing ore, comprising a step of biological leaching whereby minerals are subjected in reactors in cascade arrangement, wherein the temperature is maintained between 75°C and 85°C, to the action of a bacterial culture, which comprises a thermophilic bacterium of the *Sulfolobus* type, leading to solution of the copper, characterized in that, during said biological leaching step:

- the treatment is uninterrupted,
- the medium containing the bacterial culture is continuously mechanically agitated to ensure oxygenation thereof and suspension of the solid elements,
- the solid mass proportion of the culture medium is maintained above 10%.

The bacterial culture used is a novel

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culture which is in fact a mixture of bacteria capable of catalyzing oxidation of the sulphides and which presents an optimal growth temperature included between 75°C and 85°C. This bacterial culture has been progressively adapted in order to improve its tolerance to copper. Such an adaptation was made by successive transplantings on a substrate of chalcopyrite, in the course of which the concentrations of copper in solution were progressively and artificially increased. Such an adaptation made it possible to render these bacteria capable of developing in media of which the concentration of copper is of the order of 50 g/l.

The sulphur containing minerals are preferably furnished to the culture medium in the form of a sulphur containing concentrate presenting a granulometry d80 less than one hundred micrometers.

According to the invention, a pH included between 1.2 and 1.6 is maintained in the biological leaching reactors, particularly by addition of calcium carbonate.

The step of biological leaching will be followed by a second step during which, in a first phase the pulp issuing from the biological leaching reactors is admitted in precipitation reactors in which the iron is eliminated by provoking a precipitation of jarosite, by addition of calcite, and the solution is maintained at a pH less than 3, and the neutralized pulp is admitted in a decanter and a part of the solids is made to recirculate at the head of the precipitation reactors.

In a second phase of this form of embodiment, the liquid issuing from the decanter is admitted in

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neutralization reactors in which a pH of the order of 3.5 is maintained, particularly by an addition of calcite, so as to entrain only a minimum of copper, and one proceeds with filtration of the pulp obtained.

5 In a third phase of this form of embodiment, the aqueous phase of the liquid coming from filtration is admitted in a unit of extraction by organic solvent in which it is subjected to the action of an extracting product, so as to transfer the copper of the aqueous phase into the organic phase, the operational conditions are modified so as to transfer the copper of the organic phase of the extracting
10 product into a pure aqueous phase.

In a variant embodiment of the invention, the outlet of the extraction unit will be placed in communication with the inlet of the precipitation reactors, so as to cause part of the raffinate collected at this outlet to be recirculated by causing it to traverse said reactors again with a high flowrate with respect to the flowrate
15 of the pulp coming from the biological leaching reactors, so as to provoke a dilution of the aqueous solution subjected to extraction, up to a concentration of copper of the order of about 10 g/l, i.e. up to a value corresponding to the possible extraction of the copper in an extraction unit.

The present invention also has for an object a device for continuously
20 treating copper sulphide containing ores of the type comprising biological leaching means in which the minerals are subjected to the action of a bacterial culture leading to the solution of the copper, at

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a high concentration, of the order of 40 g/l, followed by the means for precipitating the iron contained in this solution, by addition of calcium carbonate, followed by means of extraction by organic solvent, characterized in that the outlet of the means of extraction by solvent is in communication with the inlet of the precipitation means, so as to cause a part of the raffinate collected at that outlet to recirculate, by causing it to traverse the precipitation means again with a high flowrate with respect to the flowrate of the solution at the outlet of the biological leaching means, so as to provoke a dilution of the aqueous solution subjected to extraction, up to a concentration of copper less than that existing at the outlet of the biological leaching means and preferably of the order of 10 g/l.

According to the invention, the reactors used during the biological leaching step comprise means making it possible to channel the gaseous fluid which traverses them towards condensation means.

A form of embodiment of the present invention will be described hereinafter, by way of non-limiting example, with reference to the accompanying drawing, in which:

The single Figure schematically shows the different steps of the method according to the invention.

According to the invention, the bacteria are placed in culture in stirred and aerated reactors 1 which are continuously supplied with sulphur containing minerals put in the state of pulp. This pulp is transferred from one reactor to the other by overflow. The culture medium, which is constituted by the sulphur containing minerals and the bacterial culture,

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is supplied with nutritive elements which are indispensable for the growth of the micro-organisms of the culture, whose concentrations have been optimized in order to allow a good growth thereof.

5 Of course, the reactors may be arranged in a configuration other than in cascade, and the transfer of the pulp from one reactor to the other may be effected by means other than an overflow, and in particular by pumping means, whether they be mechanical or with carrier gas effect (so-called "Air lift" systems).

10 Furthermore, it is known from the prior state of the art that the biological constitution of the external membranes of thermophilic bacteria is such that these bacteria prove to be relatively fragile as to the physical stresses of attrition that they are likely to have to undergo and which are associated with the presence of solid particles in solution when the rates of solid (expressed in percentage by
15 mass) are greater than 1%.

It is also known that these bacteria are sensitive to the shear stresses that they undergo, particularly when they are in the presence of mechanical stirring means.

20 According to the invention, the bacterial culture has also been subjected to an adaptation intended to increase the percentage of rate of solid used. To that end, the rates of solid of the solution placed in the presence of the bacterial culture have been increased, by successive stages, and the bacteria have been able to tolerate

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mass solid rates of the order of 10% to 15%.

It is known that the implementation of such a process necessitates an oxygenation of the culture medium which, in the present case, will be ensured by an injection of air at the bottom of the vat. In the event, during the process, of the concentration of dissolved oxygen proving to be insufficient, the injected air might be enriched with pure oxygen so as to improve the transfer of the oxygen towards the solution and thus promote oxidation of the sulphur containing mineral substrate. This injected air may also be enriched with carbon dioxide which constitutes the carbon substrate of this type of bacteria.

The stirring to which the bacterial culture is subjected during reaction is a mechanical stirring which is obtained with the aid of an electric motor connected to a vertical rotating shaft provided with so-called "mobile stirring" elements. A first mobile stirring element called "turbine" is disposed in low position of the shaft and is constituted in known manner by a disc of which the lower face comprises multiple radial plates which, during rotation of the disc, ensure an action of shear provoking dispersion of the air injected in the bacterial culture. The second mobile element is disposed in the upper part of the reactor and is constituted by a propeller. This mobile element presents good pumping characteristics and thus promotes the mixture and homogenization of the culture medium. The person skilled in the art will be able to optimize such stirring means so as to ensure an optimal development of the bacterial culture.

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It is ascertained that, surprisingly, the thermophilic bacterial culture used was thus in a position to withstand relatively energetic stirring means and high shear effect.

5 Furthermore, as a function of the nature of the mineral substrate used, steps will be taken to maintain the pH of the culture medium at a value preferably included between 1.2 and 1.6, and this by well mastering the various operational conditions. However, it will be noted that the value of the pH may be regulated, particularly in the event of drop of the latter, at values less than 1.2, by the
10 controlled addition of calcium carbonate, of which the dissolution will, furthermore, contribute carbon dioxide.

In order to reduce the losses of water due to evaporation in the reactors, which may be translated by a non-controlled increase in the concentration of the elements in solution capable of disturbing the development of the culture, closed
15 reactors will be used in which the emerging air flow will traverse condensation means. If such losses are not reduced sufficiently in this way, either a punctual addition of water can be made or a supply of nutritive solution without addition of concentrate can be effected, and this for a period of time adapted to the loss of water ascertained.

20 In a particularly interesting form of embodiment of the invention, a condensation system may be used intended to orientate the flow of air in order to position means for on-line analysis of the gases emerging from the reactors. Such means may thus make it possible to obtain, in real time, information on the state of the

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bacterial culture and thus to ensure complete follow-up of the treatment device.

The first step of the method of treatment according to the invention, namely the step of biological leaching, being terminated, a pulp is obtained at the outlet of the reactors which contains, in addition to the copper in solution that it is desired to isolate, at a concentration of about 40 g/l, diverse chemical products which are either dissolved in the liquid phase, or in solid form and in particular the residue of non-degraded ore, gypsum and ferric iron hydroxides.

The following steps of the method will therefore consist firstly, during an intermediate step, in separating these various components in order to extract, during a subsequent step of electrolysis, the metal copper from the purified solution.

In the course of this intermediate step, the iron will firstly be eliminated. To that end, the pulp issuing from the bank 1 of biological leaching reactors is admitted in a bank 5 of a plurality of reactors in which calcite is introduced at 6. In effect, it is known that the iron which is mainly available in its oxidized form Fe^{+3} is neutralized by the calcite which provokes a precipitation of compounds of jarosite type (i.e. a precipitate containing iron, sulphate, and a counter cation that may be H_3O^+ , Na^+ , K^+ , or NH_4^+) of hydroxides and of gypsum.

Now, it is known that the precipitation of jarosite, which is particularly interesting, since, on the one hand, it allows a lesser consumption of calcite and, on the other hand,

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the solid precipitates obtained present facilities of filtration much more interesting than the compounds of hydroxide type, is largely promoted by a relatively high temperature (which is the case of the pulp issuing from biological leaching) and that this type of precipitate is stable at low pH's (less than 3). The pH will therefore be monitored in the reactors in order to maintain it at a value less than 2.8, and this by monitoring the addition of calcite.

The installation will comprise, downstream of bank 5, in the reactors of which the jarosite precipitates, a decanter 7 which presents an outlet 8 connected to the inlet 4 of the bank 5 and which makes it possible to cause a part of the solids to recirculate "at the head of neutralization" so that, by germination, the growth of crystals will be promoted. In this way, not only the speeds of growth of the minerals, but also the characteristic of good capacity for filtration of the solids are thus improved. Here it is question of parameters which are particularly interesting in an industrial installation, as they intervene directly on the dimensioning and therefore on the cost thereof.

The rest of the iron in solution is then eliminated, in the form of hydroxides, by means of a bank 11 constituted by neutralization reactors, disposed downstream of the decanter 7, in which a pH of 3.5 is maintained by addition of calcite at 12, so as to entrain by coprecipitation only a minimum of copper. A band filter system 13 collects the pulp at the outlet 10 of the bank 11 and makes it possible to ensure a solid/liquid separation.

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Such a filtration system is constituted in known manner by a band on which the pulp is admitted and which is stretched between drums which ensure its drive in rotation. Suction means are applied through the band and a cake of increasing thickness is obtained during the displacement thereof, which is extracted at 14 after it has undergone one or more washing operations.

This solution is then sent in 16 in a unit 17 for extraction by organic solvent. It is known that such a unit is formed by a plurality of mixers/decanter in which is introduced the solution to be treated, a mixture constituted by specific extracting products and a diluent. By reason of the different affinity of the copper for the aqueous phase and for the organic phase which depends on the operational conditions, it is possible (under determined operational conditions) to transfer the copper in the organic phase then, by changing the operational conditions (contacting of the organic phase with an aqueous solution rich in sulphuric acid), to return the copper in a pure aqueous solution so as to be able then to ensure recovery of the metal copper by electrolysis. During this operation, the impurities that may pollute the cathodes during electrolysis have been eliminated.

A reactant marketed by the firm HENKEL under the Trademark "LIX" will preferably be used as extracting product.

However, it will be noted that the selective extraction of the copper with the extracting products being a

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chemical reaction of exchanges between proton and cation, the result is that, for each cuprous ion extracted, two protons are released by the extracting product. This production of acid brings about a decrease of the pH in the aqueous phase (raffinate). Now, by reason of the considerable rates of solid of the pulp issuing from the biological leaching step, the quantity of copper in solution is high and the mass of acid thus generated during the extraction induces a decrease in the pH to values which hinder extraction, and even render it impossible. It is therefore necessary to take steps for the pH to be maintained at a value compatible with that allowing extraction.

Furthermore, it has been ascertained that the first step of the method, namely the biological leaching step, delivers a pulp presenting a considerable concentration of copper, close to 40 g/l, and it is known that the operation of extraction makes it possible to extract only concentrations of copper of the order of 10 g/l at these pH values.

Of course, it might be possible to work at less high solid rates during the biological leaching step, but such treatment means would in that case impose, for an identical quantity of copper produced, that biological leaching reactors of much greater volume be available, which would have the drawback of increasing the complexity, the dimensions and the cost of the installation.

A form of embodiment of the invention, which represents a first solution allowing these drawbacks to be overcome, will be described hereinafter with reference to the single Figure.

In this form of embodiment, the outlet 19 of the extraction unit 17 is placed in communication with

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the inlet 4 of the bank 5, so as to cause a part of the raffinate collected at that outlet 19 to be recirculated by causing it to traverse the bank 5 again. In this way, upon passage therein, the addition of calcite, which is effected at 6, has the effect of increasing the pH of the solution, so as to compensate the decrease thereof due to the emission of the H^+ ions during extraction. Furthermore, by adjusting the flowrate of recirculation Q2, which is high with respect to the flowrate Q1 of the pulp admitted in the bank 5 coming from the biological leaching bank 1, a dilution of the aqueous solution subjected to extraction is provoked, to a concentration of the order of about 10 g/l, i.e. to a value corresponding to the possible extraction in an extraction unit 17.

This form of embodiment is particularly interesting insofar as it ensures extraction of the copper from a pulp with a high concentration of copper by using one single extraction unit and without additional use of devices intended to raise the pH.

In a second form of embodiment of the invention, recirculation of a part of the raffinate will not be effected, and successive means will be arranged making it possible, on the one hand, to raise the pH of the solution after extraction, and, on the other hand, to extract this solution of modified pH, until the 40 g/l of copper contained in the starting solution have been extracted.

It will then remain to carry out the last step of the method according to the invention during which, in an electrolytic cell 20,

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electrolysis of the raffinate recuperated at the end of extraction will be effected in order to recuperate the metal copper.

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CLAIMS

1. Method for treating copper sulphide containing ore, comprising a step of biological leaching whereby the minerals are subjected in reactors (1) in cascade
5 arrangement, wherein the temperature is maintained between 75°C and 85°C, to the action of a bacterial culture, which comprises a thermophilic bacterium of the Sulfolobus type, leading to solution of the copper, characterized in that, during said biological leaching step:

- the treatment is uninterrupted,

10 - the medium containing the bacterial culture is continuously mechanically agitated to ensure oxygenation thereof and suspension of the solid elements,

- the solid mass proportion of the culture medium is maintained above 10%.

2. Method according to Claim 1, characterized in that the sulphur containing
15 minerals are supplied to the culture medium in the form of a sulphur containing concentrate presenting a granulometry d80 less than one hundred micrometers.

3. Method according to one of the preceding Claims, characterized in that the bacterial culture used was previously subjected to an adaptation, by successive
20 transplants on a substrate, particularly of chalcopyrite, by progressively and artificially increasing the concentrations of the copper in solution, in order to render it able to develop in media of which the mass concentrations of copper are of the order of 50 g/l.

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4. Method according to one of the preceding Claims, characterized in that a pH included between 1.2 and 1.6 is maintained in the biological leaching reactors (1).

5 5. Method according to Claim 4, characterized in that the pH is maintained at the desired value by addition of calcium carbonate.

6. Method according to one of the preceding Claims, characterized in that all along the phase of biological leaching, the physiological state of the bacterial culture is monitored with the aid of means for on-line analysis of the gases emerging from the reactors.

7. Method according to one of the preceding Claims, characterized in that the step of biological leaching is followed by a second step during which, in a first phase:

- the pulp issuing from the biological leaching reactors (1) is admitted in

15 precipitation reactors (5) in which the iron is eliminated by provoking a precipitation of jarosite, by addition of calcite, and the solution is maintained at a pH less than 3,

- the neutralized pulp is admitted in a decanter (7) and a part of the solids is made to recirculate at the head of the precipitation reactors (5).

20 8. Method according to Claim 7, characterized in that, in a second phase:

- the liquid issuing from the decanter (7) is admitted in neutralization reactors (11) in which a pH of the order of 3.5 is maintained, particularly by an addition of calcite, so as to entrain only a minimum of copper,

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- one proceeds with filtration of the pulp obtained.

9. Method according to Claim 8, characterized in that, in a third phase,

- the aqueous phase of the liquid coming from filtration is admitted in a unit

5 (17) of extraction by organic solvent in which it is subjected to the action of an extracting product, so as to transfer the copper of the aqueous phase into the organic phase,

- the operational conditions are modified so as to transfer the copper of the organic phase of the extracting product into a pure aqueous phase.

10 10. Method according to one of Claims 7 to 9, characterized in that the outlet (19) of the extraction unit (17) is placed in communication with the inlet (4) of the precipitation reactors (5), so as to cause part of the raffinate collected at this outlet (19) to be recirculated by causing it to traverse said reactors (5) again with a high flowrate (Q2) with respect to the flowrate (Q1) of the pulp coming from

15 the biological leaching reactors (1), so as to provoke a dilution of the aqueous solution subjected to extraction, up to a concentration of copper of the order of about 10 g/l, i.e. up to a value corresponding to the possible extraction of the copper in an extraction unit (17).

11. Device for continuously treating copper sulphide containing ores of the

20 type comprising biological leaching means (1) in which the minerals are subjected to the action of a bacterial culture leading to the solution of the copper, at a high concentration, of the order of 40 g/l, followed by the means (5) for precipitating

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the iron contained in this solution, by addition of calcium carbonate, followed by means (17) of extraction by organic solvent, characterized in that the outlet (19) of the means (17) of extraction by solvent is in communication with the inlet (4) of the precipitation means (5), so as to cause a part of the raffinate collected at that outlet (19) to recirculate, by causing it to traverse the precipitation means (5) again with a high flowrate (Q2) with respect to the flowrate (Q1) of the solution at the outlet of the biological leaching means (1), so as to provoke a dilution of the aqueous solution subjected to extraction, up to a concentration of copper less than that existing at the outlet of the biological leaching means (1) and preferably of the order of 10 g/l.

12. Device according to Claim 11, characterized in that the reactors used during the biological leaching step comprise means for channeling the gaseous fluid which traverses them, towards condensation means.

METHOD AND DEVICE FOR CONTINUOUS TREATMENT OF COPPER
SULPHIDE CONTAINING ORE BY BIOLOGICAL LEACHING

5

ABSTRACT

The invention concerns a method and a device for treating copper sulphide containing ore, comprising a step of biological leaching whereby the minerals are subjected in reactors (1) in cascade arrangement, wherein the temperature is maintained between 75°C and 85°C to the action of a bacterial culture, which comprises a thermophilic bacterium of the Sulfolobus type, leading to solution heat treating of the copper. The method is characterized in that, during said biological leaching step, the treatment is uninterrupted, the medium containing the bacterial culture being continuously mechanically agitated to ensure oxygenation thereof, and suspension of the solid elements, and the solid mass proportion of the culture medium is maintained above 10%.

Translator's Note: The following legend appears in the single Figure:

- 20 FIRST STEP BIOLOGICAL LEACHING
- SECOND STEP PRECIPITATING IRON
ELIMINATING IRON IN SOLUTION AND FILTERING
EXTRACTION
- 25 THIRD STEP ELECTROLYSIS

1/1

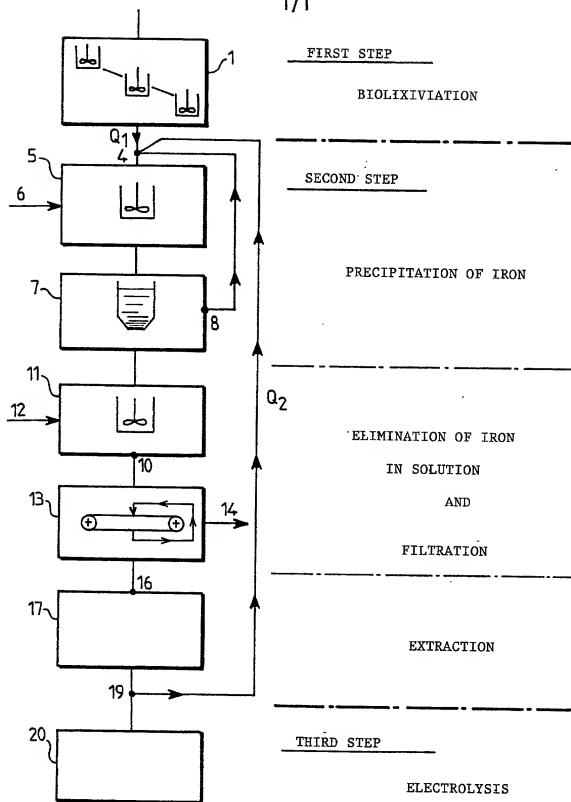


FIG.1

COMBINED DECLARATION AND POWER OF ATTORNEY

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the specification of which: *(check one)*

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Full name of sole or first inventor: Dominique MORIN
 (given name, family name)

Inventor's signature [Signature] Date 7/02/02

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 (given name, family name)

Inventor's signature _____ Date _____

Residence: Coventry CV4 7AL, Great Britain Citizenship: Great Britain

Post Office Address: Department of Biological Sciences, Gibbet Hill
Coventry CV4 7AL, Great Britain

Full name of third joint inventor, if any: Dietger KÖPPL
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Inventor's signature _____ Date _____

Residence: Düsseldorf, Germany Citizenship: Germany

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Full name of third joint inventor, if any: **Werner SCHWAB**
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Full name of third joint inventor, if any: **Christopher BONNEY**
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Inventor's signature _____ Date _____

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Post Office Address: **13 Chipping Hill, Witham**
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the specification of which: *(check one)*

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Full name of sole or first inventor: **Dominique MORIN**
(given name, family name)

Inventor's signature _____ Date _____

Residence: **Paris Cedex 15, France**

Citizenship: **France**

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Full name of second joint inventor, if any: **Paul NORRIS**
(given name, family name)

Inventor's signature *P. Norris* _____ Date **15.02.02**

Residence: **Coventry CV4 7AL, Great Britain GB**

Citizenship: **Great Britain ✓**

Post Office Address: **Department of Biological Sciences, Gibbet Hill**
Coventry CV4 7AL, Great Britain

Full name of third joint inventor, if any: **Dietger KÖPPL**
(given name, family name)

Inventor's signature _____ Date _____

Residence: **Düsseldorf, Germany**

Citizenship: **Germany**

Post Office Address: **Henkelstrasse 67**
D-40191 Düsseldorf, Germany

Full name of third joint inventor, if any: **Werner SCHWAB**
(given name, family name)

Inventor's signature _____ Date _____

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Inventor's signature _____ Date _____

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(given name, family name)

Inventor's signature _____ Date _____

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Citizenship: **Great Britain**

Post Office Address: **Department of Biological Sciences, Gibbet Hill
Coventry CV4 7AL, Great Britain**

Full name of third joint inventor, if any: **Dietger KÖPPL**
(given name, family name)

Inventor's signature *[Signature]* Date 14.2.2002

Residence: **Düsseldorf, Germany**

Citizenship: **Germany** ✓

Post Office Address: **Henkelstrasse 67
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Inventor's signature _____ Date _____

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Inventor's signature _____ Date _____

Residence: **Düsseldorf, Germany**

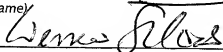
Citizenship: **Germany**

Post Office Address: **Henkelstrasse 67
D-40191 Düsseldorf, Germany**

4 Full name of third joint inventor, if any: Werner SCHWAB

1260 (given name, family name)

Inventor's signature



Date

02/15/02Residence: Düsseldorf, GermanyCitizenship: GermanyPost Office Address: **Henkelstrasse 67**
D-40191 Düsseldorf, GermanyFull name of third joint inventor, if any: **Christopher BONNEY**
(given name, family name)

Inventor's signature

Date

Residence: **Essex CM8 2DE, Great Britain**Citizenship: **Great Britain**Post Office Address: **13 Chipping Hill, Witham**
Essex CM8 2DE, Great Britain

COMBINED DECLARATION AND POWER OF ATTORNEY

As a below named inventor, I hereby declare that

My residence, post office address and citizenship are as stated below next to my name.

I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled:

METHOD AND DEVICE FOR CONTINUOUS TREATMENT OF COPPER SULPHIDE CONTAINING ORE BY BIOLOGICAL LEACHING

the specification of which: *(check one)*

REGULAR OR DESIGN APPLICATION

☐ is attached hereto.

☐ was filed on _____ as application Serial No. _____ and was amended on _____ (if applicable).

PCT FILED APPLICATION ENTERING NATIONAL STAGE

☒ was described and claimed in International application No. PCT/FR00/02471 filed on September 7, 2000 and as amended on _____ (if any).

I hereby state that I have reviewed and understand the contents of the above-identified specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose information which is material to patentability as defined in Title 37, Code of Federal Regulations, §1.56.

PRIORITY CLAIM

I hereby claim foreign priority benefits under 35 USC 119 of any foreign application(s) for patent or inventor's certificate listed below and have also identified below any foreign application for patent or inventor's certificate having a filing date before that of the application on which priority is claimed.

PRIOR FOREIGN APPLICATION(S)

Country	Application Number	Date of Filing (day, month, year)	Priority Claimed
French	99/11178	7 September 1999	Yes

(Complete this part only if this is a continuing application.)

I hereby claim the benefit under 35 USC 120 of any United States application(s) listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States application in the manner provided by the first paragraph of 35 USC 112, I acknowledge the duty to disclose information which is material to patentability as defined in Title 37 Code of Federal Regulations §1.56 which became available between the filing date of the prior application and the national or PCT international filing date of this application:

(Application Serial No.)

(Filing Date)

(Status--patented, pending, abandoned)

POWER OF ATTORNEY

The undersigned hereby authorizes the U.S. attorney or agent named herein to accept and follow instructions from Cabinet Guin & Bruder as to any action to be taken in the Patent and Trademark Office regarding this application without direct communication between the U.S. attorney or agent and the undersigned. In the event of a change in the persons from whom instructions may be taken, the U.S. attorney or agent named herein will be so notified by the undersigned.

As a named inventor, I hereby appoint the registered patent attorneys represented by Customer No. 000466 to prosecute this application and transact all business in the Patent and Trademark Office connected therewith, including: Robert J. PATCH, Reg. No. 17,355, Andrew J. PATCH, Reg. No. 32,925, Robert F. HARGEST, Reg. No. 25,590, Benoît CASTEL, Reg. No. 35,041, Eric JENSEN, Reg. No. 37,855, Thomas W. PERKINS, Reg. No. 33,027, and Roland E. LONG, Jr., Reg. No. 41,949,

c/o YOUNG & THOMPSON,
Second Floor,
745 South 23rd Street,
Arlington, Virginia 22202.



Address all telephone calls to Young & Thompson at 703/521-2297. Telefax: 703/685-0573.

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Full name of sole or first inventor: **Dominique MORIN**
 (given name, family name)

Inventor's signature _____ Date _____

Residence: **Paris Cedex 15, France** Citizenship: **France**

Post Office Address: **Tour Mirabeau, 39-43, quai André Citroën**
F-75739 Paris Cedex 15, France

Full name of second joint inventor, if any: **Paul NORRIS**
 (given name, family name)

Inventor's signature _____ Date _____

Residence: **Coventry CV4 7AL, Great Britain** Citizenship: **Great Britain**

Post Office Address: **Department of Biological Sciences, Gibbet Hill**
Coventry CV4 7AL, Great Britain

Full name of third joint inventor, if any: **Dietger KÖPPL**
 (given name, family name)

Inventor's signature _____ Date _____

Residence: **Düsseldorf, Germany** Citizenship: **Germany**

Post Office Address: **Henkelstrasse 67**
D-40191 Düsseldorf, Germany

Full name of third joint inventor, if any: **Werner SCHWAB**
(given name, family name)

Inventor's signature _____ Date _____

Residence: **Düsseldorf, Germany**

Citizenship: **Germany**

Post Office Address: **Henkelstrasse 67**
D-40191 Düsseldorf, Germany

FIFTH

5-00 Full name of third joint inventor, if any: **Christopher BONNEY**
(given name, family name)

Inventor's signature _____ Date 9/2/02

Residence: **Essex CM8 2DE, Great Britain GBN**

Citizenship: **Great Britain** ✓

Post Office Address: **13 Chipping Hill, Witham**
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